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# Erbium distribution in single crystal YAG fibers grown by laser-heated pedestal growth technique

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## ABSTRACT

Single crystal (SC) yttrium aluminum garnet (YAG,  $\text{Y}_3\text{Al}_5\text{O}_{12}$ ) as a host material has the ability to be doped with high concentrations of  $\text{Er}^{3+}$  ions. We utilize this ability to grow a 50%  $\text{Er}^{3+}$  doped YAG SC fiber, which was inserted into a SC YAG tube. This rod-in-tube was used as a preform in our laser-heated pedestal growth (LHPG) apparatus to grow a fiber with a radial distribution of  $\text{Er}^{3+}$  ions. The work shows that there is a distribution of  $\text{Er}^{3+}$  ions from their fluorescence and two different techniques were used to measure the index of refraction.

**Keywords:** Laser Heated Pedestal Growth, YAG, single-crystal fibers, infrared fiber optics, rare-earth dopants

## 1. INTRODUCTION

### 1.1 Background

Several crystal growth techniques are utilized to fabricate SC fibers, the primary ones being laser heated pedestal growth (LHPG), edge-defined film-fed growth (EFG) method, and micro-pulling-down ( $\mu$ -PD) method. In this paper, the authors have grown SC fibers using the LHPG process. Primarily developed by Haggerty at MIT in the early 1970s, LHPG is a crucibleless melt technique that can yield SC of very high purity as it precludes the possibility of contamination by the crucible material. Both active and passive SC fibers can be grown by this techniques. While passive SC fibers have been examined for potential application in high power delivery systems, active SC fibers, especially when doped by rare-earth metal ions, have generated a lot of research interest as active media for high energy fiber lasers.

### 1.2 Single Crystal Fibers

Over the past few decades, SC fibers have generated interest due to their ability to handle high optical energy density. SC fibers, especially YAG SC fibers discussed in this paper, offer a wide range of advantages over glass fibers. YAG can be doped to high concentrations without leading to clustering, unlike in glass. Apart from this, YAG has excellent thermal and mechanical properties. SC YAG fibers exhibit exceptional structural integrity, owing to their high value for Young's Modulus. The melting point of crystalline YAG is above 1900°C and its thermal conductivity is about seven times more than silica glasses, offering a higher damage threshold and hence making it far superior to the latter for high power handling.<sup>1</sup> Another remarkable property of YAG crystal is its high Stimulated Brillouin Scattering (SBS) threshold. In glass fibers, at higher power, the output is severely limited by SBS losses. However, owing to their high SBS threshold, YAG fibers have the potential to deliver five times more peak power than conventional silica glass fiber.<sup>1</sup>

Unlike conventional glass fibers, crystal fibers are grown without cladding. Fabricating a crystal, non-glass, cladding for crystal fiber has been a major challenge. Over the years, several possible methods have been proposed by research groups around the world. In this work we explore the idea of an in-growth method to potentially introduce a cladding via the distribution of the  $\text{Er}^{3+}$  ions.

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## 2. METHOD

### 2.1 Method: Laser Heated Pedestal Growth (LHPG)

LHPG is a modified float-zone crystal growth technique where the source material is melted by focusing a suitable laser beam on the preform. Unlike edge-defined film-fed growth or micro-pull down processes which involve growth from a melt contained in a crucible, LHPG is an ultra-clean process as it is not susceptible to contamination from a crucible material. In the set-up, an amplitude-stabilized 30 W DC CO<sub>2</sub> laser was utilized to form a molten zone at the tip of the source rod. The seed fiber was inserted into the molten zone and the fiber was drawn by continuously pulling the seed upward, at about a speed of 1 mm per minute. In his thesis, Fejer points out that the viscosity of molten YAG limits the source-to-fiber diameter reduction to 3:1. Hence to grow smaller diameter fibers, multiple re-growths are necessary.

Figure 2 shows a schematic of the LHPG apparatus with the main components: CO<sub>2</sub> laser, reflexicon optics, source belt drive, fiber belt drive and laser micrometer. The reflexicon converts the laser beam into a collimated ring, which is then focused to a small circular region of uniform heat at the tip of the source rod with the use of a parabolic mirror. During the growth, a PID feedback loop monitors the diameter of the fiber. The laser micrometer reads the instantaneous diameter of the fiber and this is used to calculate the instantaneous fiber pulling speed by adjusting the motor velocity through the feedback loop. This feedback diameter control loop, along with the stabilized power (which reduces instability of the molten zone), helps reduce the fluctuation in fiber diameter. Since the fiber growth is a crystal growth technique, the rate of growth is very slow so that crystal defects are minimized. Also, the laser can be focused to a single point, thus only one fiber can be drawn at a time.

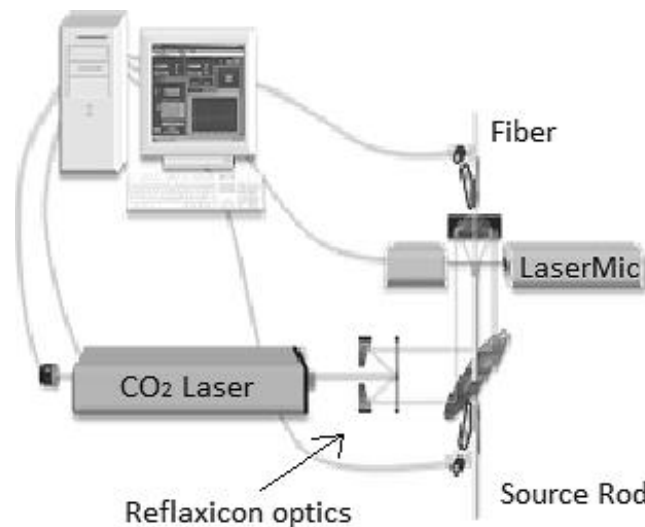


Figure 1. Schematic of LHPG.

### 2.2 CO<sub>2</sub> Laser Stability

A very critical component of our LHPG set-up is the CO<sub>2</sub> laser. It has direct impact on the molten zone, changing in size and viscosity with varying power. Thus, it is important to mediate this affect as much as possible. The direct current CO<sub>2</sub> laser used here is controlled in Labview by varying the voltage. Approximately 10 watts of power is needed for melting a 1 mm diameter source rod. As the schematic in Figure 1 shows, the laser has a 98% reflecting mirror in the back cavity. This allows control of the output power by monitoring the back power. The series of graphs on the top left of Fig. 2 show the output power of the laser, with the back transmitted power (middle), and the voltage, in this case constant voltage (bottom). The series of graphs on the right in Figure 2 is arranged the same. We note the change in min/max range from 170 mW with constant voltage to 90 mW with feed-back control on. The output power stability improves from 0.314% to 0.126% with the feedback system on. The stability is defined as the standard

deviation divided by the mean. These runs were over a duration of 10 hours. Both of these sets of data were taken after the laser warm-up period of about two hours was completed. It is also important to have an adequate water chiller, as fluctuations in water temperature will have an impact on the output power. This, along with our laser micrometer feedback loop, improve both the fiber diameter variations and fiber quality.

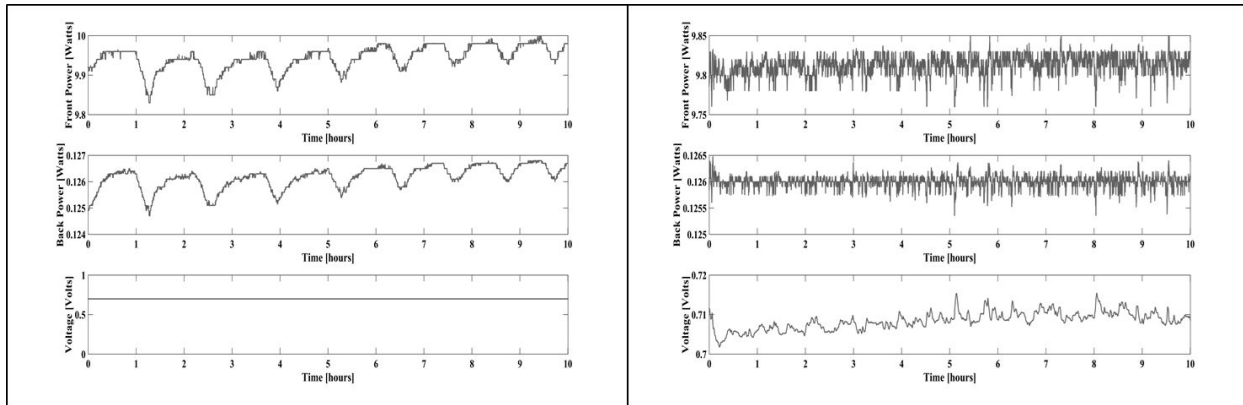


Figure 2. CO<sub>2</sub> laser under constant voltage (left) compared with Labview feed-back loop stabilizing the power (right)

### 2.3 Materials Grown by LHPG

We have grown SC YAG fibers from both SC and ceramic polycrystalline sources. In addition to pure YAG, we have grown YAG fibers doped with Er<sup>3+</sup>, Ho<sup>3+</sup>, Tm<sup>3+</sup>, Nd<sup>3+</sup>, and Yb<sup>3+</sup> – with as much as 50% Er<sup>3+</sup>, as seen in Table 1. The rare-earth doped fibers range in size from 200  $\mu\text{m}$  to 450  $\mu\text{m}$ , and growth speeds ranging from 0.5 mm/min to 2 mm/min. After only a single diameter reduction, a fiber can reach as much as ten times the length of the original source rod. Due to the similar atomic size of Er<sup>3+</sup> and Y<sup>3+</sup>, Er<sup>3+</sup> ions can be grown at high concentrations at higher velocities, unlike neodymium YAG which must be grown at very slow velocities.

Table 1. YAG materials fabricated by LHPG.

Material	Rare- Earth Dopant Concentration
Er:YAG	0.25%, 0.5%, 50%
Ho:YAG	0.5%, 4.0%
Tm:YAG	6.0%
Nd:YAG	2.5%
Yb:YAG	1%

## 3. Distribution of Erbium Ions

### 3.1 Rod-in-Tube SC Preform

A SC YAG source rod approximately 1.1 mm in diameter was initially cut into one centimeter long sections. These sections were then mechanically drilled with an inner diameter of approximately 550  $\mu\text{m}$  seen in Fig. 3, at which point a 50% Er<sup>3+</sup>:YAG fiber was inserted inside the tube section. This acts as our rod-in-tube preform, which will then be used as the source pedestal. During heated pedestal growth the Er<sup>3+</sup> ions are diffusing outward into the pure YAG, therefore the duration the material is in the melt has the largest impact, with temperature being held constant. The two factors most controllable when using constant temperature are: changing the diameter of the grown fiber and/or changing the growth speed. We grew the fiber at 380  $\mu\text{m}$  at a velocity of approximately 1 mm/min for the three different profiles that follow. These parameters could be changed to study the differences in the Er<sup>3+</sup> ions distribution.

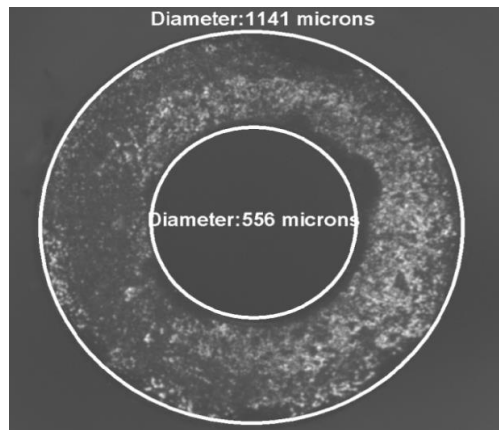


Figure 3: Image of a YAG tube, with an inner diameter of 550  $\mu\text{m}$

### 3.2 Fluorescence Profile

The 380  $\mu\text{m}$   $\text{Er}^{3+}$ :YAG fiber was thinly sliced to obtain the radial distribution using fluorescence measurements. The fiber was excited at 532 nm, and in Fig. 4 the relative radial  $\text{Er}^{3+}$  ion intensity is apparent. It is worth noting that when sampling a pure  $\text{Er}^{3+}$ :YAG fiber the fluorescence profile was uniform.

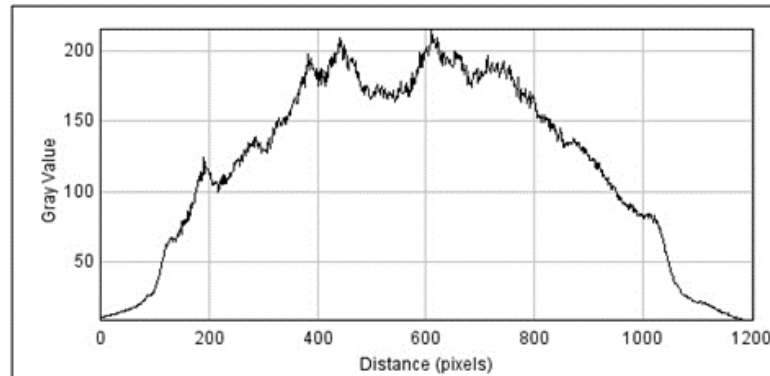


Figure 4: Fluorescence profile

### 3.3. Index Profiling

A Fresnel reflection apparatus was designed for radial index of refraction measurements to be on a cross section of the fiber. The reflections were obtained from a laser operating at 592 nm with a spatial resolution of 4  $\mu\text{m}$ . At 592 nm the index of pure YAG is 1.832.<sup>6</sup> Due to YAG's high refractive index it has a high reflectance of 8.6% at normal incidence. Once again, for a fiber grown under the same conditions, we show in Fig. 5 a relative radial index change from the center of the fiber to the edge. The samples were polished with 0.1  $\mu\text{m}$  alumina polishing paper. The noise in Fig. 5 is due to polishing defects, such as scratches.

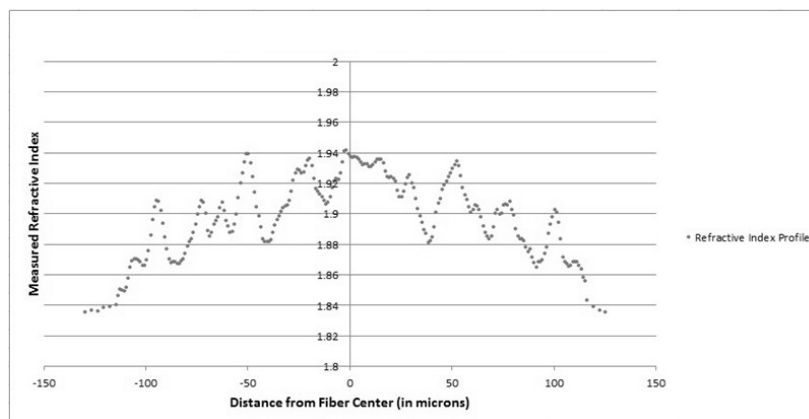


Figure 5: Refractive index profile using a Fresnel reflection apparatus

The third means of investigating the radial distribution was by using transverse interferometry to measure the refractive index of the rod-in-tube fiber. This work was done by Interfiber Analysis and, as seen in Fig. 6, shows an increase of approximately 0.005 in refractive index at the center. Their technique uses index matching fluids and measures the optical path length difference. This results in interference fringes which can be used to compute the optical path length and obtain a fiber's refractive index profile from the fringes. The sharp spikes at the peripheral edges are a result of the index fluid used not exactly matching the index of YAG. The index fluids over 1.8 are hazardous and could not be used. Thus, at the operating wavelength of 950 nm, the oil had an index of 1.79. Some accuracy is lost due to this mismatch, but there is certainly a relative change in the index at the center.

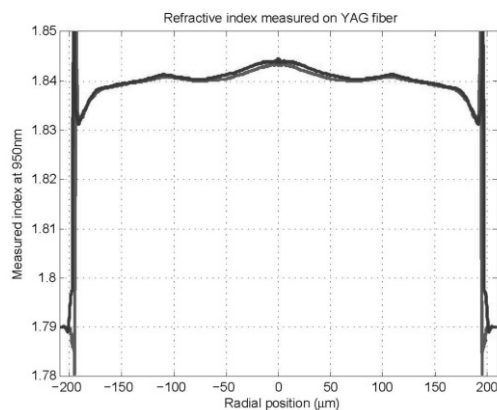


Figure 6: Refractive index profile using interferometry

#### 4. CONCLUSION

This work does show that a distribution of  $\text{Er}^{3+}$  ions does appear to have an effect on the radial refractive index. This aspect could be used to form a graded index fiber that could act as a cladding. It is also possible to use a co-doped system; one to change the radial index, while the other could be the lasing ion.

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